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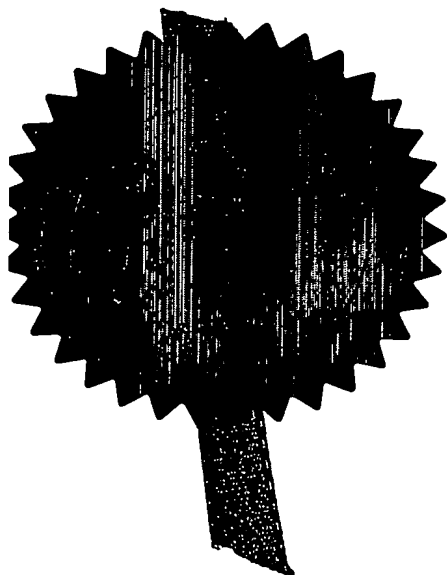
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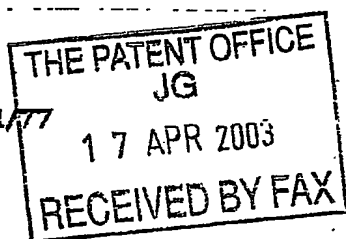
*Stephen Hordley*

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AA 1649 GB

## 2. Patent application number

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0308944.8

17APR03 E001200-1 001091  
P01/7700 0.00-0308944.83. Full name, address and postcode of the or of each applicant (*underline all surnames*)JOHNSON MATTHEY PUBLIC LIMITED COMPANY  
2-4 COCKSPUR STREET  
TRAFALGAR SQUARE  
LONDON SW1 5BQPatents ADP number (*if you know it*)

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## 4. Title of the invention

METHOD OF DECOMPOSING NITROGEN DIOXIDE

5. Name of your agent (*if you have one*)

ANDREW DOMINIC NUNN

"Address for service" in the United Kingdom  
to which all correspondence should be sent  
(*including the postcode*)JOHNSON MATTHEY TECHNOLOGY CENTRE  
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Claim(s)	4
Abstract	0
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AA 1649

**METHOD OF DECOMPOSING NITROGEN DIOXIDE**

5 The present invention relates to a method of decomposing nitrogen dioxide ( $\text{NO}_2$ ) in a gas mixture, and in particular it relates to a method of decomposing  $\text{NO}_2$  in a  $\lambda > 1$  gas mixture.

10 Exhaust gases from internal combustion engines comprise a mixture of pollutants including carbon monoxide (CO), unburned hydrocarbons (HC), nitrogen oxides ( $\text{NO}_x$ ) and particulate matter (PM). The  $\text{NO}_x$  component can comprise nitrogen monoxide (NO) and nitrogen dioxide ( $\text{NO}_2$ ). The level of these pollutants in exhaust gases from internal combustion engines it is permissible to exhaust to atmosphere is regulated by legislation. Such legislation can be met by engine design, engine management and/or exhaust gas aftertreatment, and typically a combination of all three measures.

15 A prior art exhaust system primarily for treating diesel exhaust comprises an oxidation catalyst for oxidising NO in the exhaust gas to  $\text{NO}_2$  and a downstream filter for trapping PM. A process for treating diesel PM that uses this arrangement is described in EP 0341382 or US patent no. 4,902,487, both of which are incorporated herein by reference. The process comprises passing an exhaust gas, such as a diesel exhaust gas, including PM and NO unfiltered over an  
20 oxidation catalyst to convert the NO to  $\text{NO}_2$ , collecting soot on the filter and combusting the collected soot by reaction with the  $\text{NO}_2$ . This technology is commercially available as Johnson Matthey's Continuously Regenerating Trap or CRT<sup>®</sup>. Combustion of the PM in  $\text{NO}_2$  results in CO and NO, with a potential side-reaction leading to the complete reduction of the  $\text{NO}_2$  to  $\text{N}_2$  as described in SAE 890404.

25 An advantage of this process is that it is possible to combust diesel soot at temperatures of up to  $400^\circ\text{C}$ , whereas combustion of diesel soot in oxygen occurs at about  $500^\circ\text{C}$ . This is significant since diesel exhaust gas is generally cooler than exhaust gas from gasoline engines and soot would accumulate on the filter causing back-pressure problems in the system if the  
30 process relied on combustion of soot in oxygen.

A problem with the process describe in EP 0341382 is that, as exhaust emission legislation has tightened since the publication of that application, legislative bodies have begun to discuss limiting the amount of  $\text{NO}_2$  it is permissible to exhaust to atmosphere.  $\text{NO}_2$  is toxic

and can cause headaches, dizziness and nausea in low doses. It also has an objectionable smell. If there is insufficient PM on the filter to react with  $\text{NO}_2$  generated over the oxidation catalyst or the temperature of the exhaust gas is below a preferred range for combustion of PM in  $\text{NO}_2$ ,  $\text{NO}_2$  can slip past the filter and be undesirably exhausted to atmosphere.

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This problem is particularly acute when internal combustion engines are used in confined spaces, such as mines, where vehicles are used to drill for, load, and transport mined material to the surface. Many mining operations generate particulate matter, and so exhaust aftertreatment systems comprising filters for reducing the levels of PM emitted are being considered. Furthermore, explosives used to blast rock to recover a desired ore can generate  $\text{NO}_2$ . Accordingly, it would be an advantage to reduce the exhaust gas emissions of both PM and  $\text{NO}_2$  to the atmosphere in closed environments to improve the health and safety of miners.

10

One potential solution to this problem is described in EP 758713, where in one embodiment, an exhaust system comprises an optionally platinum-based oxidation catalyst and a diesel particulate filter (DPF) in the CRT<sup>®</sup> configuration and a  $\text{NO}_x$  absorbent downstream of the DPF. The  $\text{NO}_x$  absorbent can comprise platinum for oxidising NO to  $\text{NO}_2$  in  $\lambda > 1$  exhaust gas compositions, rhodium for reducing  $\text{NO}_x$  to  $\text{N}_2$  in  $\lambda < 1$  exhaust gas compositions and at least one substance selected from alkali metals such as potassium and caesium, alkali-earth metals such as barium and calcium; and rare-earth metals such as lanthanum for absorbing the  $\text{NO}_2$  and storing it as the nitrate. Catalyst compositions comprising platinum, rhodium and a  $\text{NO}_x$  absorbent material are typically called  $\text{NO}_x$  traps.

15

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In our PCT/GB02/004750 filed 21<sup>st</sup> October 2002, we describe an exhaust system for an internal combustion engine comprising a catalyst, such as a platinum-based catalyst, for oxidising NO to  $\text{NO}_2$  when the exhaust gas composition is  $\lambda > 1$ ; and a filter disposed downstream of the NO oxidation catalyst, i.e. in the CRT<sup>®</sup> configuration. The filter can comprise an oxidation catalyst such as platinum and/or palladium, rhodium and a  $\text{NO}_x$  absorbent material, such as any of those described in EP 758713 above. The filter component of this arrangement per se is described in Japanese patent application no. 2722987.

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A disadvantage of exhaust systems comprising  $\text{NO}_x$  traps is that the  $\text{NO}_x$  absorbent material has a finite capacity and in order to maintain  $\text{NO}_x$  conversion in the system to meet the appropriate emissions legislation, the  $\text{NO}_x$  absorbent material needs to be, at least partially,

regenerated. In practice this is done by controlling the composition of the exhaust gas so that transiently it is shifted to  $\lambda < 1$  from normal  $\lambda > 1$  operation periodically throughout a drive cycle. Lambda composition of the exhaust gas can be changes, for example, by adjusting the period and duration of fuel injection into one or more cylinders, retarding fuel injection timing in one or more cylinders or injecting HC into the exhaust system downstream of the engine. Such arrangements requires very careful and complicated control of the engine and fuel injection timing and means to estimate or measure remaining  $\text{NO}_x$  absorption capacity in the  $\text{NO}_x$  trap.

10 We have investigated catalysts for decomposing  $\text{NO}_2$  in the exhaust gas of internal combustion engines and we have discovered, very surprisingly, a catalyst with particular activity for this reaction in  $\lambda > 1$  exhaust gas conditions.

15 According to a first aspect, the invention provides a method of decomposing nitrogen dioxide ( $\text{NO}_2$ ) in a  $\lambda > 1$  gas mixture, which method comprising contacting the gas mixture with a supported platinum group metal (PGM) catalyst, wherein the PGM consists of rhodium.

Potentially, the method according to the first aspect of the invention can be used to treat gas mixtures including  $\text{NO}_2$  generated by any chemical, e.g. industrial, process. However, in a particular embodiment, the method is for treating an exhaust gas mixture derived from combustion of a hydrocarbon fuel, such as diesel fuel, gasoline fuel, natural gas (NG) or liquid petroleum gas (LPG).

25 According to a second aspect, the invention provides an exhaust system for an internal combustion engine, which system comprising a supported platinum group metal (PGM) catalyst for decomposing  $\text{NO}_2$  in a  $\lambda > 1$  exhaust gas mixture, wherein the PGM consists of rhodium.

30 An important feature of the invention is that the support on which the rhodium is supported is devoid of other platinum group metals. Furthermore, the supported rhodium is not mixed with other support(s) supporting non-rhodium PGMs. Put another way, the supported rhodium is segregated from non-rhodium PGMs present in the exhaust system. According to one embodiment, the supported rhodium catalyst is disposed downstream of an oxidation catalyst comprising at least one PGM, preferably at least one of platinum and palladium. In one

embodiment, the oxidation catalyst is on a filter, e.g. an upstream end thereof, whereas the supported rhodium catalyst is on a downstream end of a filter.

A particular embodiment according to the invention is a NO oxidation catalyst and a filter in the CRT<sup>®</sup> configuration, wherein a downstream end of the filter comprises the supported rhodium catalyst. An upstream end of the filter can comprise an oxidation catalyst, such as platinum and/or palladium-based catalyst, for reducing the combustion temperature of the PM trapped therein. A filter including an oxidation catalyst for combusting PM therein is often referred to as a catalysed soot filter (CSF).

The method and exhaust system of the present invention are different from those described in EP 758713 and PCT/GB02/004750 in that in the prior art, the rhodium is a component in a NO<sub>x</sub> trap composition, i.e. it is associated with other catalysts such as platinum and NO<sub>x</sub> absorbent materials such as potassium, caesium and calcium. Furthermore, it is understood from the description of e.g. EP-A-1079084 discussed in PCT/GB02/004750 that any rhodium present would be on the upstream end of the filter.

According to a further embodiment of the invention, the oxidation catalyst in the CSF can be associated with a NO<sub>x</sub> absorbent material, such as any of those described in connection with EP 758713 above. In one embodiment, natural fluctuations in exhaust gas temperature and exhaust gas composition are used to regenerate the NO<sub>x</sub> absorbent material. In a further aspect, the exhaust system comprises a means for controlling the composition of the exhaust gas so that transiently it is shifted to  $\lambda < 1$  from normal  $\lambda > 1$  operation periodically throughout a drive cycle. Methods of achieving such control of exhaust gas composition are described above and can be controlled, in use, by a suitably programmed engine control unit comprising a microprocessor.

The filter can be any suitable for the intended purpose, and can be made from metal e.g. Fecralloy<sup>™</sup>, or ceramic, such as of cordierite. In one embodiment, the filter is a wall-flow filter, e.g. of a ceramic material of suitable pore size distribution.

Rhodium loading on the support can be from 0.1 to 5.0 wt% based on the total weight of the support. In one embodiment, the rhodium loading is from 0.25 to 0.2 wt% based on the total weight of the support. We have used 0.5wt% rhodium in the Examples with beneficial results.

The support can be any suitable for the purpose such as alumina, ceria, zirconia, titania, silica, chromia, or a mixture, composite oxide or mixed oxide of any two or more thereof. In certain embodiments, we believe it may be advantageous to use a support material with an oxygen storage function e.g. ceria or composite or mixed oxides comprising ceria, such as ceria-zirconia.

"Composite oxide" as defined herein means a largely amorphous oxide material comprising oxides of at least two elements which are not true mixed oxides consisting of the at least two elements.

The composite oxide or mixed oxide can be silica-alumina, e.g. a zeolite such as  $\beta$ -zeolite, ZSM-5 or Y-zeolite, a silica-zirconia, alumina-zirconia, alumina-chromia, alumina-ceria or ceria-titania.

The support can be doped with at least one of lanthanum, barium, cerium, tungsten, silica and manganese to improve the stability of the support to high temperature ageing, act as a promoter in the decomposition of  $\text{NO}_2$  and/or improve the oxygen storage capacity of the system, particularly in the case of manganese.

In one embodiment, the exhaust system comprises a bypass means for bypassing the supported rhodium catalyst when the composition of the exhaust gas mixture is  $\lambda \leq 1$  and control means, in use, for controlling the bypass means to bypass the supported rhodium catalyst with at least a proportion of an exhaust gas flowing in the system when the composition of the exhaust gas mixture is  $\lambda \leq 1$ . In this way, the  $\text{NO}_2$  reduction feature of the supported rhodium catalyst can be limited to  $\lambda > 1$  conditions. However, in exhaust systems including a  $\text{NO}_x$  absorbent material upstream of the supported rhodium catalyst it is desirable to maintain the supported rhodium catalyst "in line" so that the known reduction activity of rhodium can be utilised to reduce  $\text{NO}_x$  in the exhaust gas to  $\text{N}_2$  in  $\lambda < 1$  conditions.

The control means for controlling the bypass means can comprise an engine control unit comprising a suitably programmed microprocessor.



According to a preferred embodiment, the invention provides an exhaust system for a diesel engine, which system comprising a platinum group metal, preferably platinum, -based oxidation catalyst disposed upstream of a ceramic diesel particulate wall-flow filter (DPF), wherein a downstream end of the filter comprises a supported platinum group metal (PGM) catalyst for decomposing  $\text{NO}_2$  in a  $\lambda > 1$  exhaust gas, wherein the PGM consists of rhodium.

According to a third aspect, the invention provides an internal combustion engine comprising an exhaust system according to the invention. Such engine can be fuelled by any suitable fuel such as diesel fuel, gasoline fuel, natural gas (NG) or liquid petroleum gas (LPG).

According to a fourth aspect, the invention provides a vehicle, such as a mining vehicle, comprising an engine according to the third aspect of the invention.

It is believed that the supported rhodium catalyst described herein, in addition to catalysing the reduction of  $\text{NO}_2$ , may also catalyse the reduction of  $\text{SO}_3$  to  $\text{SO}_2$  in exhaust gas conditions and can be used where such reaction is desirable, e.g. to reduce the amount of  $\text{SO}_3$ -derived particulate observed in diesel drive cycles.

In order that the invention may be more fully understood, the following Examples are provided by way of illustration only and with reference to the accompanying drawings, in which:

Figure 1 is a graph showing % $\text{NO}_2$  conversion as a function of temperature ( $^{\circ}\text{C}$ ) for supported platinum and rhodium catalysts;

Figure 2 is a graph showing steady state % $\text{NO}_2$  decomposition as a function of temperature as a function of temperature ( $^{\circ}\text{C}$ );

Figure 3 is a graph showing NO production over various catalyst samples as a function of temperature ( $^{\circ}\text{C}$ );

Figure 4 is a graph showing the effect of gaseous components on  $\text{NO}_2$  decomposition over  $0.5\text{Rh}/\text{Al}_2\text{O}_3$ ;

Figure 5 is a graph showing the effect of gaseous components on NO production over  $0.5\text{Rh}/\text{Al}_2\text{O}_3$ .

### Example 1

A series of catalysts were analysed on a simulated catalyst activity test (SCAT) gas rig for their abilities of decomposing  $\text{NO}_2$  below its equilibrium concentration in a reaction mixture that contains 200ppm  $\text{NO}_2$ , 12%  $\text{O}_2$ , 4.5%  $\text{CO}_2$ , 4.5%  $\text{H}_2\text{O}$  and 20ppm  $\text{SO}_2$ . The catalysts were ramped in the reaction mixture to 500°C and then steady state conversion at 50°C temperature interval was measured during cool down.

Figure 1 shows the steady state  $\text{NO}_2$  decomposition over 2.5Pt/ $\text{Al}_2\text{O}_3$  (2.5wt% platinum by total weight of the particulate support), 0.5Rh/ $\text{Al}_2\text{O}_3$  (0.5wt% rhodium by total weight of the particulate support) and a blank reactor. The blank reactor did not show much  $\text{NO}_2$  conversion even at high temperature, although thermodynamic predictions would indicate that  $\text{NO}_2$  is unstable above 350°C and should decompose to NO. It would appear that the  $\text{NO}_2$  decomposition reaction is a slow process and thermodynamic equilibrium is not achieved without a catalyst. Pt could decompose  $\text{NO}_2$  to NO to the expected equilibrium concentration. The Rh catalyst, however, achieves a surprisingly higher  $\text{NO}_2$  decomposition conversion.

Figure 2 shows compares the activity of 0.5Rh/ $\text{Al}_2\text{O}_3$  to decompose  $\text{NO}_2$  against three further catalyst systems: 85Mn:15Zr (see US patent no. 5,283,041); 5wt% copper based on the total weight of zeolite ZSM-5 particulate support; and 5wt% silver based on the total weight of particulate alumina support. The exhaust gas composition is the same as for Figure 1 tests.

Figure 3 shows NO production over various catalyst samples as a function of temperature (°C) for the same catalysts featured in Figure 2 and in addition particulate bulk ceria.

### Example 2

Figures 4 and 5 show, respectively, the effect of gaseous components on  $\text{NO}_2$  decomposition over 0.5Rh/ $\text{Al}_2\text{O}_3$  and the effect of gaseous components on NO production over 0.5Rh/ $\text{Al}_2\text{O}_3$ . The synthetic gas compositions were: (triangles) 200ppm  $\text{NO}_2$ , 12%  $\text{O}_2$ ,  $\text{N}_2$  balance; (diamonds) 200ppm  $\text{NO}_2$ , 12%  $\text{O}_2$ , 4.5%  $\text{CO}_2$ , 4.5%  $\text{H}_2\text{O}$  and 20ppm  $\text{SO}_2$ ,  $\text{N}_2$  balance; and (squares) 200ppm  $\text{NO}_2$ , 12%  $\text{O}_2$ , 4.5%  $\text{CO}_2$ , 4.5%  $\text{H}_2\text{O}$  and 20ppm  $\text{SO}_2$ , 100ppm CO, 100ppm  $\text{C}_3\text{H}_8$ ,  $\text{N}_2$  balance.

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It can be seen that the addition of CO and  $C_3H_6$  - both components of internal combustion engine exhaust gases e.g. diesel exhaust gas - improves the low temperature conversion of  $NO_2$  to NO.

**CLAIMS:**

1. A method of decomposing nitrogen dioxide ( $\text{NO}_2$ ) in a  $\lambda > 1$  gas mixture, which method comprising contacting the gas mixture with a supported platinum group metal (PGM) catalyst, wherein the PGM consists of rhodium.  
5
2. A method according to claim 1, wherein the rhodium is loaded on the support at from 0.1 to 5.0 wt% based on the total weight of the support.
- 10 3. A method according to claim 2, wherein the rhodium loading is from 0.25 to 0.2 wt% based on the total weight of the support.
- 15 4. A method according to claim 1, 2 or 3, wherein the support is alumina, ceria, zirconia, titania, silica, chromia, or a mixture, composite oxide or mixed oxide of any two or more thereof.
- 20 5. A method according to claim 4, wherein the composite oxide or mixed oxide comprises silica-alumina, a silica-zirconia, alumina-zirconia, alumina-chromia, alumina-ceria, ceria-titania or ceria-zirconia.
6. A method according to claim 1, 2 or 3, wherein the support is alumina.
- 25 7. A method according to any preceding claim, wherein the support is doped with at least one of lanthanum, barium, cerium, tungsten, silica and manganese.
8. A method according to any preceding claim, wherein the gas is produced by a chemical process.
- 30 9. A method according to claim 8, wherein the chemical process comprises combustion of a hydrocarbon fuel.
10. A method according to claim 9, wherein the hydrocarbon fuel is diesel fuel, gasoline fuel, natural gas (NG) or liquid petroleum gas (LPG).

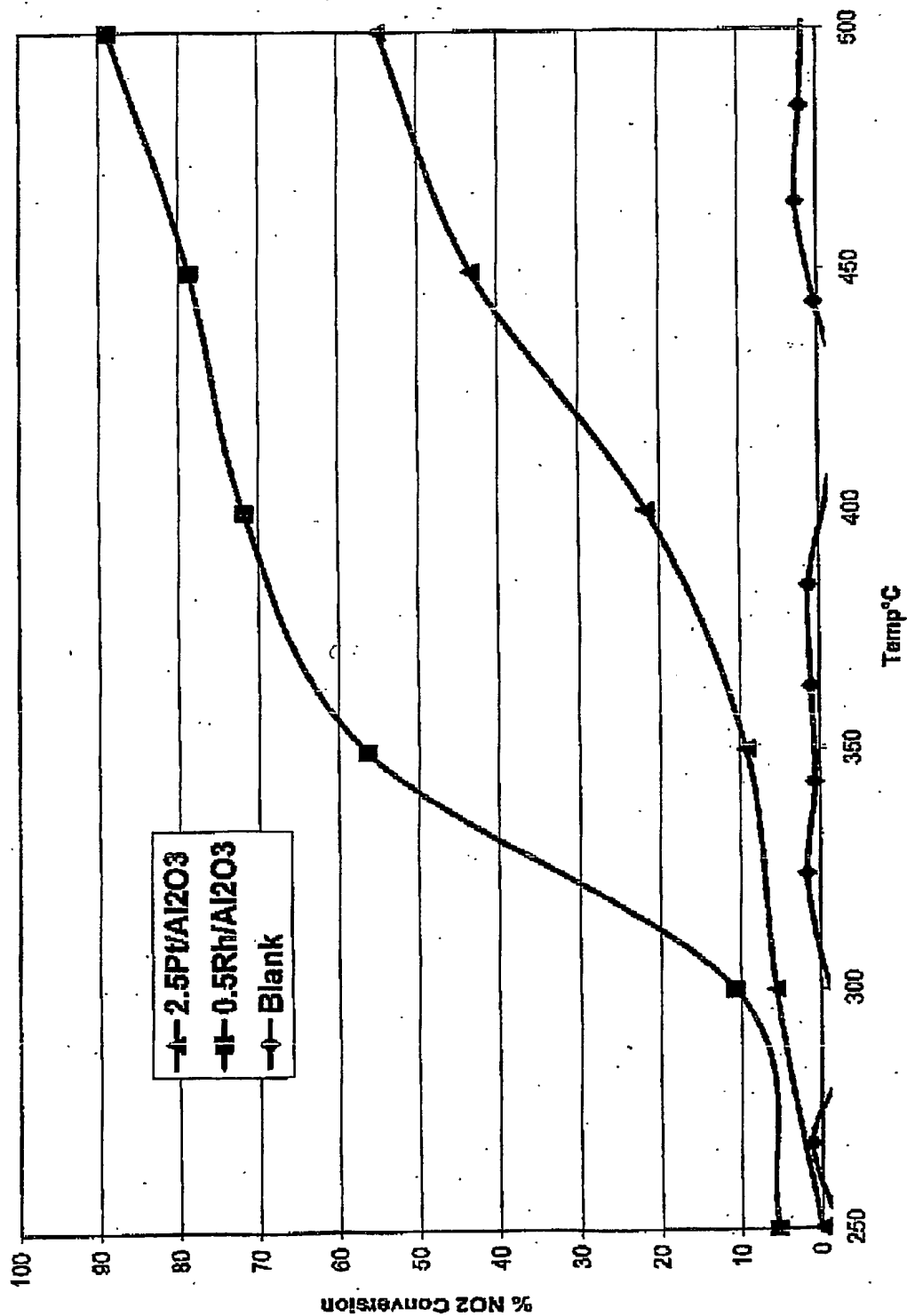
11. An exhaust system for an internal combustion engine, which system comprising a supported platinum group metal (PGM) catalyst for decomposing  $\text{NO}_2$  in a  $\lambda > 1$  exhaust gas mixture, wherein the PGM consists of rhodium.
- 5 12. An exhaust system according to claim 11, wherein the supported rhodium catalyst is disposed downstream of an oxidation catalyst comprising at least one PGM, preferably at least one of platinum and palladium.
- 10 13. An exhaust system according to claim 11 or 12, wherein the supported rhodium catalyst is disposed on a downstream end of a filter.
14. An exhaust system according to claim 13 when appendant on claim 12, wherein an upstream end of the filter comprises the PGM oxidation catalyst.
- 15 15. An exhaust system according to claim 14, wherein the PGM oxidation catalyst is associated with a  $\text{NO}_x$  absorbent material.
16. An exhaust system according to claim 13, wherein the filter is a wall-flow filter.
- 20 17. An exhaust system according to any of claims 11 to 16, wherein the rhodium is loaded on the support at from 0.1 to 5.0 wt% based on the total weight of the support.
18. An exhaust system according to claim 17, wherein the rhodium loading is from 0.25 to 0.2 wt% based on the total weight of the support.
- 25 19. An exhaust system according to any of claims 11 to 18, wherein the support is alumina, ceria, zirconia, titania, silica, chromia, or a mixture, composite oxide or mixed oxide of any two or more thereof.
- 30 20. An exhaust system according to claim 19, wherein the composite oxide or mixed oxide comprises silica-alumina, a silica-zirconia, alumina-zirconia, alumina-chromia, alumina-ceria, ceria-titania or ceria-zirconia.

21. An exhaust system according to claim any of claims 11 to 18, wherein the support is alumina.
22. An exhaust system according to any of claims 11 to 21, wherein the support is doped with at least one of lanthanum, barium, cerium, tungsten, silica and manganese.
23. An exhaust system according to any of claims 11 to 22, further comprising bypass means for bypassing the supported rhodium catalyst when the composition of the exhaust gas mixture is  $\lambda \leq 1$  and control means, in use, for controlling the bypass means to bypass the supported rhodium catalyst with at least a proportion of an exhaust gas flowing in the system when the composition of the exhaust gas mixture is  $\lambda \leq 1$ .
24. An exhaust system according to claim 23, wherein the control means comprises an engine control unit comprising a suitably programmed microprocessor.
25. An exhaust system for a diesel engine, which system comprising a platinum group metal, preferably platinum, -based oxidation catalyst disposed upstream of a ceramic diesel particulate wall-flow filter (DPF), wherein a downstream end of the filter comprises a supported platinum group metal (PGM) catalyst for decomposing  $\text{NO}_2$  in a  $\lambda > 1$  exhaust gas, wherein the PGM consists of rhodium.
26. An internal combustion engine comprising an exhaust system according to any of claims 11 to 25.
27. An engine according to claim 26 fuelled with diesel fuel, gasoline fuel, natural gas (NG) or liquid petroleum gas (LPG).
28. A vehicle, such as a mining vehicle, comprising an engine according to claim 26 or 27.
29. A method of decomposing nitrogen dioxide ( $\text{NO}_2$ ) in a  $\lambda > 1$  gas substantially as described herein.
30. An exhaust system substantially as described herein.

31. An internal combustion engine substantially as described herein.

1/5

Figure 1





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Figure 2

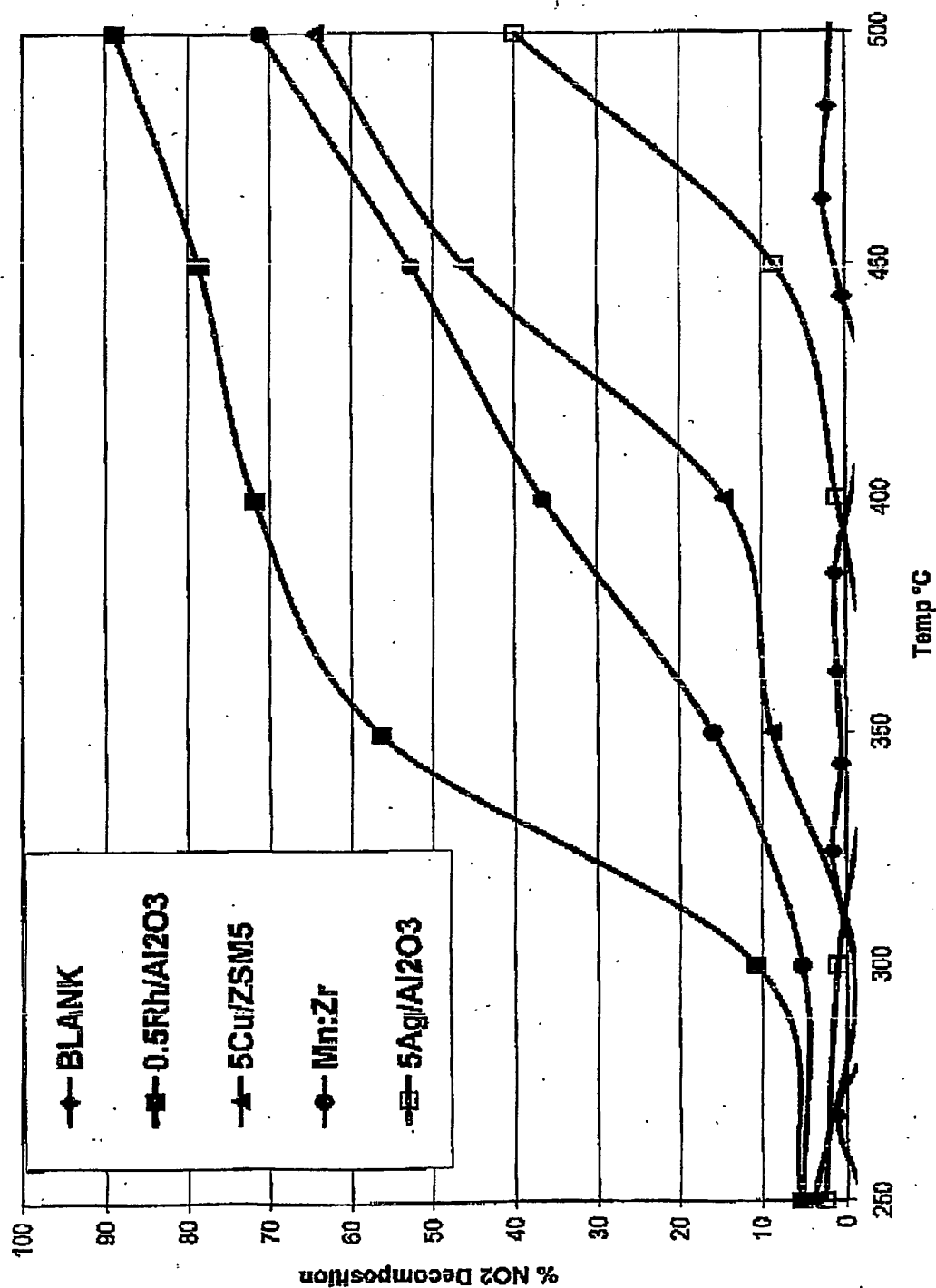
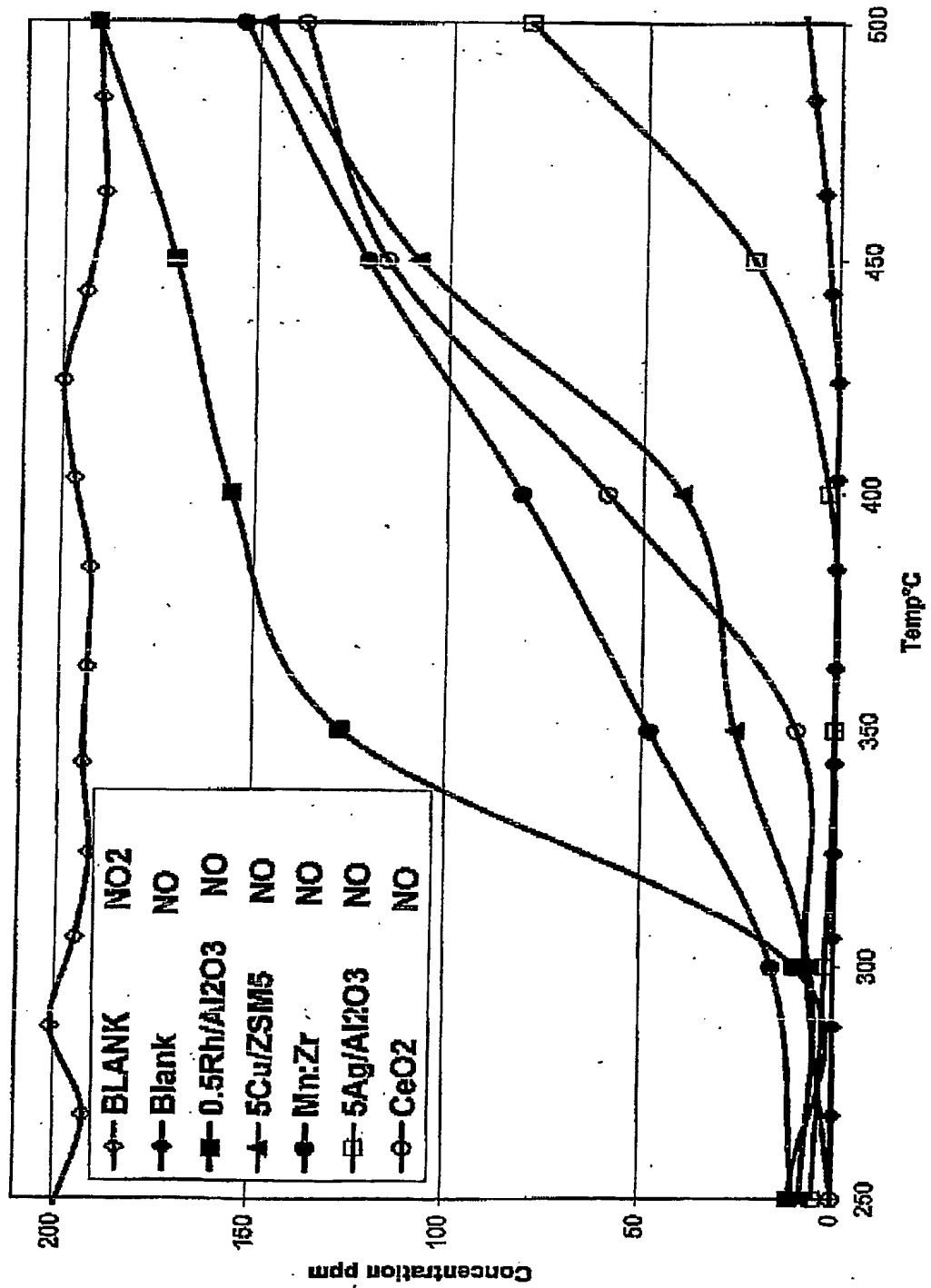
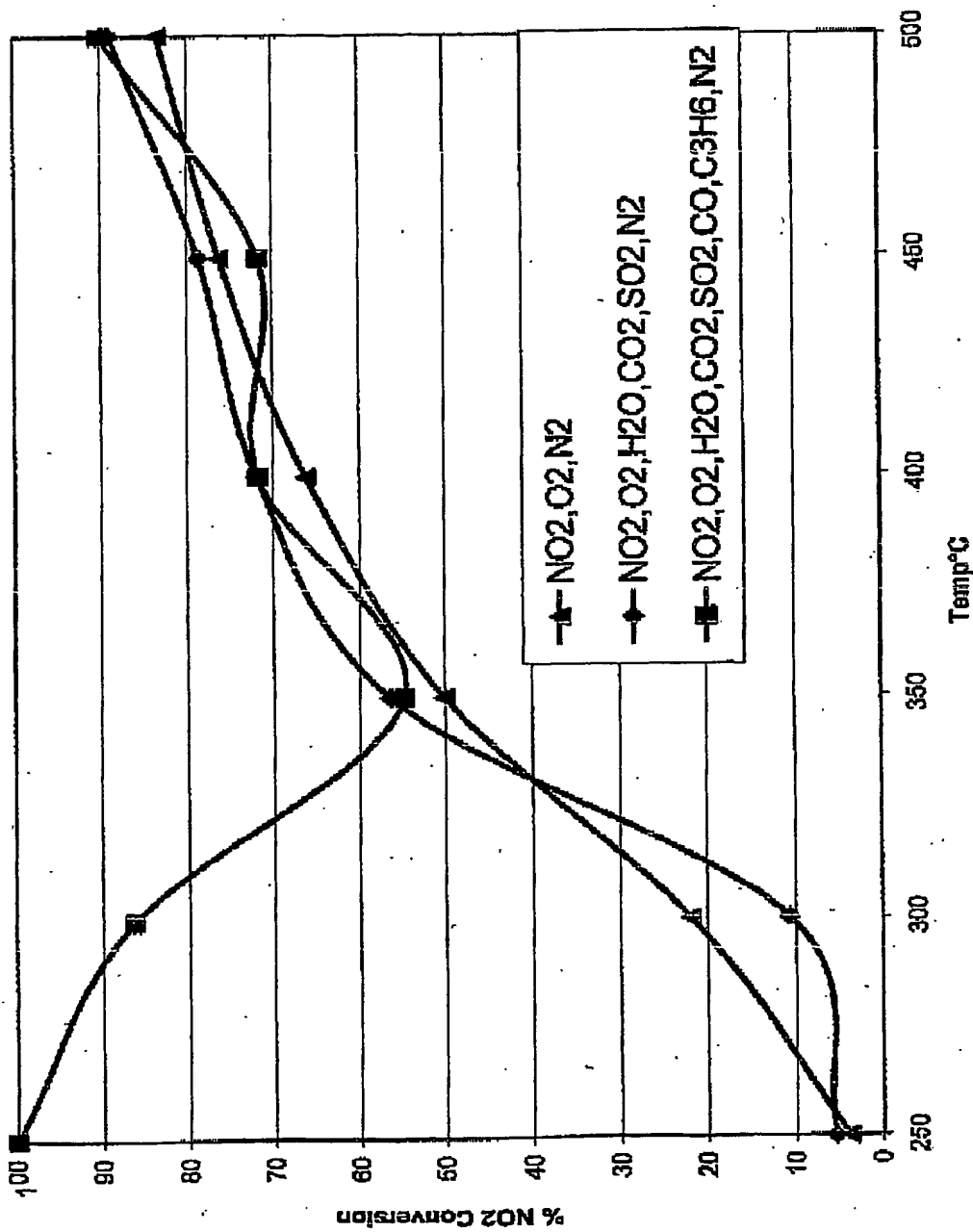


Figure 3



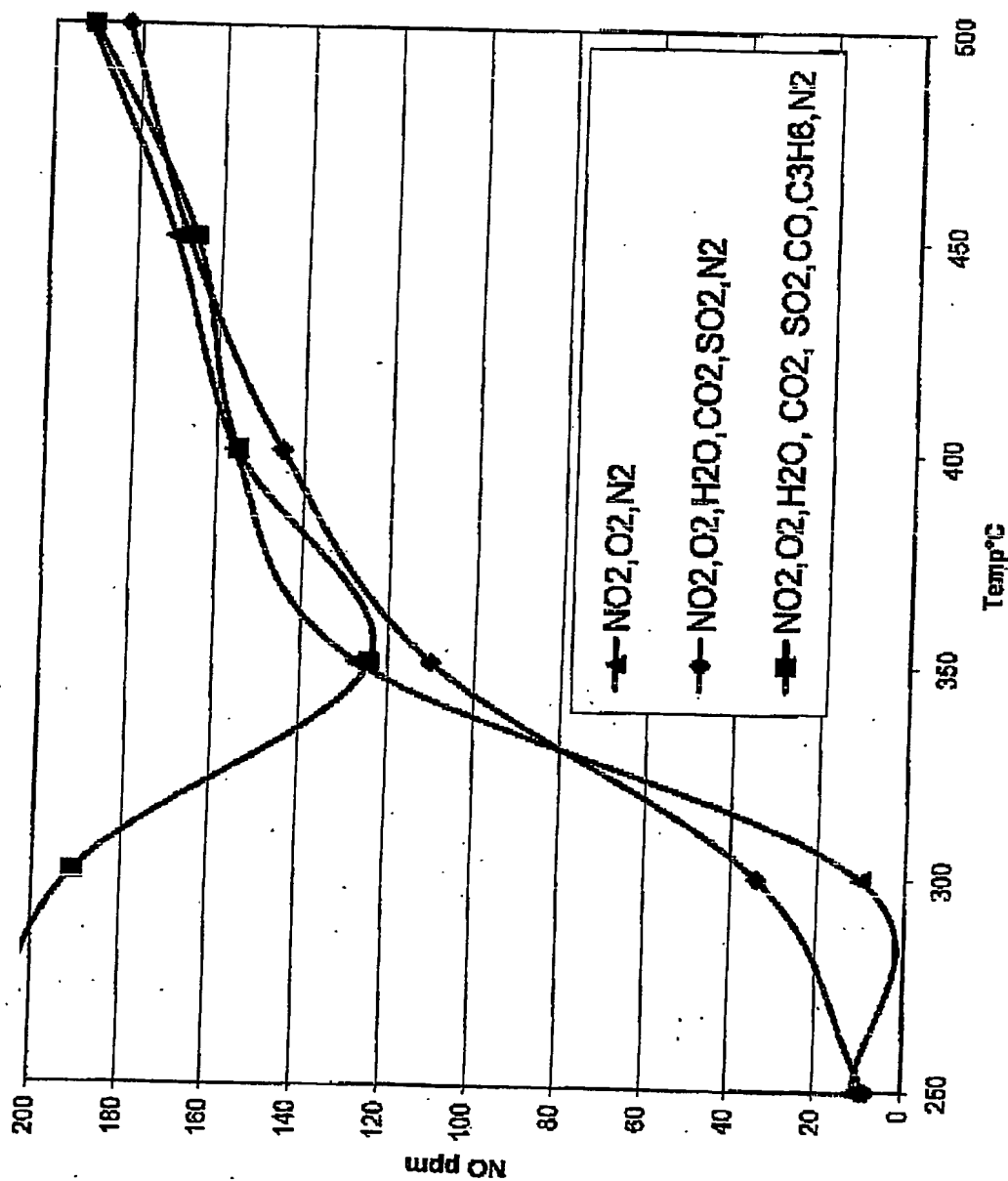
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Figure 4



5/5.

Figure 5



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